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[PCD 3: Petroleum, Lubricants and their Related Products]

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Indian Standard

MOLYBDENUM DISULPHIDE DISPERSION IN OIL — SPECIFICATION

UDC 621·892

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

August 1993

Price Group 2

Lubricants and Related Products Sectional Committee, PCD 4

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Lubricants and Related Products Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

Molybdenum disulphide (MoS_2) dispersion in oil is an important medium of using MoS_2 as lubricant. It has found wide application where friction reducing and extreme pressure properties are required, for example, in metal working as well as antisiege compounds or as an additive in lubricants.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

MOLYBDENUM DISULPHIDE DISPERSION IN OIL — SPECIFICATION

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for molybdenum disulphide dispersion in oil.

2 NORMATIVE REFERENCES

The following Indian Standards contain provisions which through reference in this text constitute the provisions of the standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
1447 : 1966	Methods of sampling of petroleum and its products
1448	Methods of test for petroleum and its products
13796 : 1993	Molybdenum disulphide powder for lubricants

3 REQUIREMENTS

3.1 General

The material shall be homogenous, dark fluid, free from water, grit, foreign matter and visible impurities.

3.2 Composition

The material shall consist of a stable dispersion of molybdenum disulphide in refined filtered mineral lubricating oil having a viscosity index (V.I.) 80 *Min* and kinematic viscosity at 40°C as 90 - 110 cSt and shall contain approximately 20 percent of molybdenum disulphide powder by mass conforming to IS : 13796 1993.

3.3 The material shall also comply with the requirements given in Table 1 when tested according to the methods prescribed in col 4 and 5 of Table 1.

4 KEEPING QUALITY

The material shall retain the properties described in 3 of this specification for a period not less than one year from the date of manufacture when stored in its original sealed container under cover and at ambient temperature conditions not exceeding 50°C.

5 PACKING AND MARKING

5.1 Packing

The material shall be packed as agreed to between the purchaser and the supplier.

5.2 Marking

The containers shall be marked with the following information:

a) Name of the material;
b) Net mass in the container;
c) Indication of the source of manufacture;
d) Date of manufacture;
e) Recognized trade-mark, if any; and
f) Batch number or code number.

5.2.1 The containers may also be marked with the Standard Mark.

6 SAMPLING

6.1 Representative samples of the material shall be drawn in IS 1447 : 1966.

Table 1 Requirements for Molybdenum Disulphide Dispersion in Oil

(*Clause* 3.3)

Sl No.	Characteristic	Requirement	Method of Test	
			Annex of this Standard	[P :] of IS 1448
1)	(2)	(3)	(4)	(5)
i)	Stability, percent, *Min*	50	A	—
ii)	Flash point (COC), °C, *Min*	170	—	P : 21
iii)	Kinematic viscosity at 100°C, cSt	14 to 30	—	P : 25
iv)	Acidity, inorganic, mg KOH/gm	Nil	—	P : 2 (Method C)
v)	Moisture content, percent by mass	Nil	B	—
vi)	Filtered solids content, percent by mass	20 ± 1	C	—
vii)	Size of molybdenum disulphide particles:			
	a) Greater than 5 microns in diameter, percent number, *Max*	2	D	—
	b) Residue on 63 microns IS test sieve	Nil	E	—

ANNEX A

[*Table* 1, *Item* (i)]

DETERMINATION OF STABILITY OF MOLYBDENUM DISULPHIDE IN OIL

A-1 PROCEDURE

A-1.1 Approximately 15 ml of the dispersion is transferred to each of two opposed 15-ml centrifuge tubes and centrifuged at 1 000 G's (2 300 rpm in the International Centrifuge Model SBV Size 1) for 1 h.

A-1.2 The supernatant liquid from each of the set of opposed tubes is decanted, combined and homogenized (by stirring) in a 50 ml beaker. Duplicate samples for analysis are taken from the homogenized decanted by withdrawing 6 ml with a disposable syringe and placing about 3 ml in each of two tared 100 ml beakers and then reweighing to obtain the sample mass. The analysis may then be conducted by atomic absorption without concern for any settling out of the molybdenum disulphide from the suspension.

A-1.3 The centrifuge stability is taken as the percent of molybdenum sulphide remaining in suspension after 1 h at 1 000 G's centrifuge test, that is:

$$\text{Molybdenum disulphide percent} = 100 \times \frac{\text{percent molybdenum remaining suspended}}{\text{molybdenum disulphide in original suspension}}$$

ANNEX B

[*Table* 1, *Item* (v)]

DETERMINATION OF MOISTURE CONTENT IN MOLYBDENUM DISULPHIDE DISPERSION IN OIL

B-1 GENERAL

This test is used as a check on possible water contamination.

B-2 APPARATUS

B-2.1 Silica Crucible — 50 ml capacity.

B-2.2 Bunsen Burner

B-2.3 Tripod Stand

B-3 PROCEDURE

Take approximately 25 g of dry bright stock oil (150/160) in the crucible and heat it till the oil starts boiling. Add approximately 1 ml of dispersion under test to the boiling oil in the form of drops and listen carefully for a crackling sound as the product falls into the crucible (*see* Note).

NOTE — It is recommended that this test is carried out in a fume cup-board with an efficient extractor fan.

B-4 REPORTING

Repot as 'moisture present' if crackling sound is heard or 'NIL' if no sound is recorded.

ANNEX C

[*Table* 1, *Item* (vi)]

DETERMINATION OF FILTERED SOLIDS CONTENT IN MOLYBDENUM DISULPHIDE DISPERSION IN OIL

C-1 APPARATUS

C-1.1 Gooch Crucible

C-1.2 Source of Vacuum

C-1.3 Beakers

C-1.4 Desiccator

C-1.5 Oven Thermostatically — Controlled at 120°C.

C-2 PROCEDURE

C-2.1 Sufficient product containing approximately 30 mg of colloidal solids is accurately weighed into a clean dry 150 ml beaker. 50 ml of a compatible volatile solvent (for example, butyl acetate) are gradually added with stirring until mixing is complete, 50 ml of a solvent flocculent are then added with stirring and allowed to stand until the colloid is seen to be flocculated. This may take several hours.

C-2.2 The mixture is then filtered through the tared gooch crucible under vacuum, usually by means of a pump. The filtrate shall be free of solid particles. If not, the filtrate shall be passed through the crucible until it is clean. Any solids remaining in the beaker are washed out with a further 50 ml of solvent. All washings are filtered through the Gooch crucibles.

C-2.3 The solid residue in the crucible is then washed with about 500 ml of the volatile solvent in order to remove all traces of oil. The crucible is then dried in an oven at 120°C for thirty minutes and cooled in a desiccator.

C-2.4 The crucible is then weighed and the mass of residue determined. Repeat the procedure given under **C-2.3** until a constant mass of residue is obtained (*see* Note under **C-3**).

C-3 CALCULATION

The colloidal solids content of the product is calculated as follows:

$$\text{Filtered solids content, percent by mass} = \frac{M_2 \times 100}{M_1}$$

where

M_2 = Mass in g of solids retained by the Gooch crucible, and

M_1 = Mass in g of the product taken for the test.

NOTE—It is recommended that all determinations may be performed in duplicate. A tared Gooch crucible is prepared for each duplicate.

ANNEX D

[*Table* 1, *Item* (vii) (a)]

DETERMINATION OF THE SIZE OF MOLYBDENUM DISULPHIDE PARTICLES

D-1 GENERAL

The method assesses the approximate number of molybdenum disulphide particles in size greater than 5 micron in diameter and below 5 microns.

D-2 APPARATUS

D-2.1 Optical Microscope

With a maximum magnification of at least 1 500 with provision for oil immersion.

D-2.2 Balance

D-2.3 Stirrer

D-2.4 Beaker — 150 ml capacity, tall form.

D-2.5 Paraffins — Medicinal quality.

D-3 PROCEDURE

D-3.1 Take 0·2 g of the material in a tall 150 ml beaker. Add 20 ml of medicinal paraffin and stir thoroughly to ensure uniform dispersion. Transfer one small drop of this diluted dispersion to a clearly microscope slide and cover with a slip ensuring even distribution of the suspension and removal of air pockets. Count the number of particles (t) in a total area (a) of the slide sufficiently large to contain at least 100 particles and consisting of at least 10 sub-areas chosen at random over the slide.

D-3.2 Count the number of particles (g) greater than 5 microns in diameter in an area (b) of the slide sufficiently large to contain at least 50 such particles at the same magnification. If less than 50 particles are counted when the area (b) is 100 times the area (a) the count may be discontinued.

D-4 CALCULATION

$$\text{Particles greater than 5 microns in diameter, percent} = \frac{a \times g}{b \times t} \times 100$$

ANNEX E

[*Table* 1, *Item* (vii) (b)]

DETERMINATION OF THE SIZE OF MOLYBDENUM DISULPHIDE PARTICLES (RESIDUE ON 63 MICRON IS SIEVE)

[*Adapted from IND/SL/4116* (*a*)]

E-1 PROCEDURE

E-1.1 Weigh 50 g of the material into a beaker and dilute with 100 ml of white spirit, then pass it through a 63 micron IS test sieve. Wash the sieve with white spirit until the washings are clear.

E-1.2 Dry the sieve at 105±5°C and examine for any sign of residue.

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